Effect of Composite Fabrication on the Strength of Single Crystal Al_2O_3 Fibers in Two Fe-Base Alloy Composites

by

S. L. Draper and B. J. M. Aikin

ABSTRACT

Continuous single-crystal Al_2O_3 fibers have been incorporated into a variety of metal and intermetallic matrices and the results have consistently indicated that the fiber strength had been reduced by 32 to 50% during processing.[1-4] Two iron-based alloys, FeNiCoCrAl and FeAlVCMn, were chosen as matrices for Al_2O_3 fiber reinforced metal matrix composites (MMC) with the goal of maintaining Al_2O_3 fiber strength after composite processing. The feasibility of Al_2O_3/FeNiCoCrAl and Al_2O_3/FeAlVCMn composite systems for high temperature applications were assessed in terms of fiber-matrix chemical compatibility, interfacial bond strength, and composite tensile properties. The strength of etched-out fibers was significantly improved by choosing matrices containing less reactive elements. The ultimate tensile strength (UTS) values of the composites could generally be predicted with existing models using the strength of etched-out fibers. However, the UTS of the composites were less than desired due to a low fiber Weibull modulus. Acoustic emission analysis during tensile testing was a useful tool for determining the efficiency of the fibers in the composite and for determining the failure mechanism of the composites.

I. INTRODUCTION

Alumina fiber is an attractive candidate for the reinforcement of metal and intermetallic matrix composites (MMC's and IMC's) due to its high strength and modulus, low density, and commercial availability. Al_2O_3 was predicted to be thermodynamically stable with many metals and intermetallics of interest for high temperature applications.[1, 5-6] However, continuous single-crystal Al_2O_3 fibers have been incorporated into a variety of metal and intermetallic matrices, including FeAl, NiAl, FeCrAlY, and Ni-base superalloys, and the results have consistently indicated that the fiber strength had been reduced by 32 to 50% during processing.[1-4] The loss of Al_2O_3 fiber strength during fabrication was detrimental to the composite strength at room and elevated temperatures. Four proposed mechanisms that may contribute to the fiber strength loss include reactions with the matrix, reactions with binders, residual stress-induced damage, and pressure from hot pressing.[1] In past work, the effects of matrix reactions were separated from the other three effects by sputter-coating the matrices on cleaned fibers.[1] The sputter-coated fibers were annealed using a temperature profile that simulated processing conditions. The effects of the matrix reactions appeared to dominate over the other possible mechanisms, although some reactions with binders could also be a significant factor for some systems. Y and Cr in FeCrAlY base alloys and Zr in NiAl alloys reacted with the fibers and formed grooves on the fiber surfaces. Research by Westfall[7] also indicated that high Cr levels in Fe-Ni-Cr-

Al alloys were detrimental to fiber strength. In order to improve the strength of MMC's and IMC's using continuous single-crystal Al_2O_3 fibers, the strength of the fibers must be maintained during processing by use of coatings or less reactive matrices.

Two iron-based alloys, with less reactive elements than FeCrAlY, were chosen as matrices for Al_2O_3 fiber reinforced metal matrix composites with the goal of maintaining Al_2O_3 fiber strength after composite processing. The first alloy was a modified version of Thermo-span®, a low thermal expansion alloy with a low Cr content available from Carpenter Technology Corporation (Reading, PA). Originally, powder with essentially the Thermo-span® composition, Table I, was investigated. However, severe fiber strength degradation occurred during fabrication and Nb and Ti particles adhered to the etched out fiber surfaces. Subsequently, a modified version of Thermo-span® (without Nb and Ti), designated FeNiCoCrAl, was selected as a matrix. The low thermal expansion coefficient of this alloy was also expected to be beneficial in terms of reduced residual stresses in the composite. The second alloy in this study, designated FeAlVCMn, is an alloy developed for intake valves for internal combustion engines[8]. This alloy was chosen because it had attractive strength and ductility combined with a composition significantly different than those examined previously.

The feasibility of Al_2O_3/FeNiCoCrAl and Al_2O_3/FeAlVCMn composite systems were assessed in terms of fiber-matrix chemical compatibility, interfacial bond strength, and composite tensile properties. The experimentally measured composite tensile properties were compared to predicted values. Acoustic emission (AE) instrumentation was used during room-temperature tensile tests to aid in determining the failure mechanisms of the composites. Interpretation of the AE parameters can only be done in general terms, but some correlation of AE parameters to damage modes has been established.[9,10] Results of the acoustic emission testing and microstructural analyses of the composites after tensile testing were related to possible failure mechanisms.

II. MATERIALS AND EXPERIMENTAL PROCEDURE

The powder cloth technique[11] and a tape-casting technique[12] were used to fabricate 5 cm wide by 15 cm long composites with 6-plies of continuous Al_2O_3 fibers. C-axis, single-crystal (Saphikon) Al_2O_3 fibers, 125 mm diameter, were oriented in the 0° direction (parallel to the long direction of the composite). In the powder cloth technique, the matrix powder was mixed with a poly(tetrafluoethylene) (TEFLON)[a] binder and rolled out into a clothlike sheet. The fibers were wound on a drum, and a poly(methyl methacrylate) (PMMA) binder was applied to produce a fiber mat. The powder cloth and fiber mats were stacked and the composite was consolidated in a vacuum hot press (VHP). The tape casting process consists of winding the Al_2O_3 fibers onto a drum and then casting a mixture of matrix powder, a solvent (toluene), and a copolymer binder (polyisobutylene(PIB)) and poly(methyl methacrylate))[12] onto the wound fibers.

[a]TEFLON is a trademark of E.I. Du Pont de Nemours & Co., Inc., Wilmington, De.

The fiber/matrix mats were stacked, with matrix-only plies used on the top and bottom. The composites were consolidated in a vacuum hot press.

The degradation of Al_2O_3 fiber strengths (σ_f) were determined by comparing the strengths of as-received fibers to the strengths of fibers etched from the powder cloth and tape-cast composite plates. As-received fibers were taken from the spool just before and after winding the fibers for use in the composite plates. In a previous study,[2] a group of fibers were tested in the as-received plus etched state to ensure that the etching procedure used to remove the fibers from consolidated plates was not damaging the fibers. Fibers were etched from the matrices using a solution of 33% H_2O, 33% HNO_3, and 33% HCl (by volume) at room temperature. The Al_2O_3 fibers were tensile tested at room temperature with a crosshead speed of 1.27 mm/min and a gage length of 12.7 mm. Twenty fibers were tested for every condition. The diameters of the fibers were measured using a laser micrometer. All error bars in this paper represent 95% confidence intervals assuming a Guassian distribution.

Interfacial shear strengths were measured by the fiber push-out technique at room temperature. Details of the fiber push-out technique have been described elsewhere.[13]

Monolithic tensile specimens were machined by wire electrodischarge machining, whereas composite specimens were machined by water jet cutting. Three

14 cm long, reduced gage specimens with a 15.2-mm gage length were machined from each plate. Specimen surfaces were polished with 600 grit SiC paper before testing. The specimens were tested in air at a constant crosshead speed of 0.13 mm/min. Strain was measured with an axial extensometer attached to the edges of the specimen. Acoustic emission data were collected from each composite tensile test. Two sensors, with resonant frequencies of 250 kHz, were used to eliminate noise outside the gage length and to locate failure events within the gage section, similar to previous work.[2]

Optical microscopy and scanning electron microscopy (SEM) were performed on as-fabricated and tensile-tested samples. Longitudinal metallographic specimens were polished to examine fiber cracks, and transverse sections were polished to measure the volume fraction of fibers.

III. RESULTS

A. *Microstructure*

Typical fiber distributions for both composite systems, fabricated by both powder cloth and tape casting techniques, are shown in Figure 1. Variations in fiber diameters are also evident, typical of the Saphikon process (Milford, NH). Fiber volume fractions were approximately 26 to 33%. Fiber spacing was slightly better in the tape cast composites with very few touching fibers. The matrix of the FeAlVCMn powder-cloth composites was not dense, therefore, the tensile results of this composite are not included. The matrix was fully consolidated for both alloys fabricated by the tape-casting technique. The FeAlVCMn alloy had small precipitates, which are most likely vanadium carbides based on energy dispersive spectrometer analysis (EDS). Interfacial reaction zones were not visible by scanning electron microscopy for either composite system, Figure 2.

Chemical analyses of the composite specimens were performed to determine residual binder content, Table II. Nonzero values of fluorine are indicative of incomplete TEFLON® burn-off in the powder cloth fabricated specimens. Oxygen contents could not be measured in the composites due to the presence of oxide fibers. FeAlVCMn contains C and therefore the presence of C from binder contamination was not determined.

Fiber breakage has occurred during fabrication of Al_2O_3 fiber reinforced IMC's and MMC's, with considerable variation in the quantity of the breakage from plate to plate.[2-3] The lengths of fibers etched-out of a Al_2O_3/FeAlVCMn and a Al_2O_3/FeNiCoCrAl tape-cast composite plate were measured and plotted in a histogram, Figure 3. Visual inspection and nondestructive evaluation have shown fiber breakage clustered in specific areas, typically near both ends of the 15 cm long composite. The longest fiber etched from the Al_2O_3/FeAlVCMn plate was 115 mm and the fiber length mode was

101-110 mm. The mode of fibers etched from tape-cast fabricated Al_2O_3/FeNiCoCrAl composite plates was in the range of 131-140 mm. The tape-cast fabricated Al_2O_3/FeNiCoCrAl plate had a slightly larger quantity of broken fibers compared to a Al_2O_3/FeNiCoCrAl plate fabricated by the powder cloth technique. However, the differences may be within plate to plate variations and may not reflect a difference between the fabrication techniques.

B. Etched-Out Fiber Strength

The tape-cast and powder cloth fabrication techniques resulted in similar fiber strength degradations for the FeAlVCMn matrix composites, around 34% from the as-received fiber strength, Table III. The surface of the fibers etched from the composites were examined in the SEM. Fibers etched from FeAlVCMn had fairly smooth surfaces but clusters of V-enriched particles, approximately 1 μm in diameter, adhered to the surfaces, Figure 5. The fiber's surface did not have any grooves, as was observed from fibers etched from FeCrAlY[2] or ridges, as observed from fibers etched from NiAl.[1]

The room temperature tensile strengths of the Al_2O_3 fibers etched from FeNiCoCrAl was significantly different between the powder cloth and tape cast fabricated composite plates, with averages of 2203 ± 136 and 2719 ± 250 MPa respectively (Table III). The tape cast composite processing resulted in only a 24% degradation in strength. The data is plotted on a Weibull plot in Figure 4 and the estimated Weibull moduli are listed in Table III. A significant difference between the surface of fibers etched from FeNiCoCrAl matrix composites fabricated by powder-cloth and tape-casting was observed. The surfaces of the fibers etched from the powder-cloth composite had a fine-scale dimpling of the fiber surface, Figure 6a. Occasional particles that were found had traces of Cr or contained Na, Cl, and Ca, possibly contaminates from the etching procedure. A reaction between the binders used in the powder-cloth technique, matrix, and fiber could be the cause of the dimpled fiber surface. However, a dimpled fiber surface has never been seen before with other matrices using the powder cloth technique.[1-3] The fibers etched from tape-cast FeNiCoCrAl had smooth surfaces, similar to as-received fibers, Figure 6b.

C. Interfacial Shear Strength

Fiber-matrix interfacial shear stresses were determined on the tape-cast composites using a fiber push-out technique,[13] Table IV. Typical room temperature fiber load/displacement curves are shown in Figure 7 for both composite systems. Fiber debonding,[13] t_d, is indicated by a sharp drop in load and is further verified by a spike in the acoustic emission signal. The frictional shear stresses, t_f, are also indicated on the load/displacement curves in Figure 7.

The interfacial bonds in Al_2O_3/FeNiCoCrAl were quite weak. While a drop in load indicated debonding, an accompanying spike in the acoustic emission was not always observed. The low interfacial shear strength, averaging 30 ± 3 MPa (Table IV), indicates little, if any, chemical bond between Al_2O_3 and FeNiCoCrAl. Bonding between Al_2O_3 and FeAlVCMn was considerably higher, averaging 158 ± 15 MPa.

D. FeAlVCMn and Al_2O_3/FeAlVCMn Tensile Behavior

Monolithic FeAlVCMn yielded at an average of 762 ± 23 MPa at room temperature and strain hardened to an average ultimate tensile strength (UTS) of 817 ± 29 MPa, Figure 8 and Table V. Failure occurred at an average strain of 1.20 ± 0.48%.

The powder cloth fabricated Al_2O_3/FeAlVCMn composites, which were tensile tested, contained porosity. Therefore, only the results of the Al_2O_3/FeAlVCMn composites fabricated by tape casting are reported in Table VI. The room temperature stress-strain curves, Figure 8, consisted of an initial linear region followed by a slightly curved region up to the UTS of the material, which averaged 1096 ± 231 MPa. Failure occurred at the ultimate tensile stress (UTS) at approximately 0.7 % strain. The elastic modulus of the composite averaged 238 ± 26 GPa, significantly higher than the monolithic at 163 ± 16 GPa.

The tensile tests were performed with acoustic emission (AE) sensors attached to both ends of the tensile specimens, allowing the location of AE events to be identified. Amplitude histograms of AE events were bimodal with peaks at low decibels

(42 db) and high decibels (93 db), Figure 9a. High decibel levels, greater than 90 db, are typical of fiber fractures.[2, 9-10] The lower decibel level events can be attributed to matrix yielding, matrix cracking, or fiber/matrix debonding. The location of high amplitude AE events as a function of strain is plotted in Figure 9b. The first high amplitude event occurred at 0.18% strain, above where the stress-strain curve deviated from linearity (e_{el}). At 0.3% strain, fiber fractures were occurring throughout the gage section. At 0.6% strain, fiber fractures started to concentrate 20 mm from the bottom sensor (the center of the gage section) and failure occurred at 0.72% strain (ε_f) at this location.

A polished longitudinal section of an Al_2O_3/FeAlVCMn specimen shows broken fibers close to the fracture surface and occasional fractured fibers away from the fracture surface, consistent with AE results, Figure 10. The fracture surface exhibited little fiber pullout, Figure 11a, and the matrix failed intergranularly.

E. FeNiCoCrAl and Al_2O_3/FeNiCoCrAl Tensile Behavior

Monolithic FeNiCoCrAl had lower strength than FeAlVCMn, with an average yield strength ($\sigma_{0.2\%y}$) of 254 ± 5 MPa and an UTS of 464 ± 56 MPa. FeNiCoCrAl is very ductile and extensions exceeded the limit on the tensile tests, set at 10 and 20%. The elastic modulus averaged 157 ± 27 GPa, similar to FeAlVCMn.

Even though there were significant differences in the strengths of etched-out fibers between powder-cloth and tape-cast Al_2O_3/FeNiCoCrAl, the tensile behavior of the composites was indistinguishable. Therefore, the powder-cloth and tape-cast tensile data was pooled but individual results are listed in Table VI. The room temperature stress-strain curves, Figure 8, consisted of an initial linear region followed by a slightly curved region up to the UTS of the material, which averaged 643 ± 34 MPa. At the UTS, a load drop occurred. Stress was held fairly constant as the composite elongated to 1.6% strain. Fiber breakage continued resulting in a jagged appearance in the stress-strain curves with the load eventually decreasing to zero. The UTS occurred at an average strain of 0.42 ± 0.11%. The elastic modulus of the composite averaged 234 ± 42 GPa, significantly higher than the modulus of monolithic FeNiCoCrAl.

The AE results were similar for both powder-cloth and tape-cast Al_2O_3/FeNiCoCrAl composites. The number of events was higher for this composite system but the event amplitudes had a similar bimodal distribution, Figure 12a. The high amplitude events were filtered-out and a source location plot of these events is shown in Figure 12b. The first high amplitude event occurred at 0.12% strain, just above the elastic limit of the composite. The first event was quickly followed by additional events scattered throughout the gage section of the composite. Following the load drop at 0.49% strain (ε_{UTS}), fiber fractures concentrated at 5 mm above the bottom sensor which eventually became the location of the composite failure, Figure

12b. Fibers fractures stopped at 1.5% strain, however, the composite continued to accumulate strain as the load dropped to zero. One powder-cloth fabricated composite tensile specimen did not fail and the test was stopped at 2.5 % strain. Indentations on the specimen surface, taken to be fiber fractures, were spread over a 13 mm wide region and directly correlated to the AE results.

Metallography of a fracture surface confirmed the AE results for both tape-cast and powder-cloth fabricated composites. Fibers were broken into short segments close to the fracture surface, Figure 10b, and a few fiber fractures were scattered throughout the gage section. Extensive fiber pull-out was observed on the fracture surface of the Al_2O_3/FeNiCoCrAl tensile specimens, Figure 11b.

A Weibull analysis of etched-out fiber strength and in-situ fiber strength as determined by AE was plotted, Figure 13, for both composite systems. In-situ fiber strength was calculated by multiplying the Young's modulus of the fiber (452 GPa[14]) by the strain at an AE event. Weibull plots of etched-out fiber strengths and AE determined fiber strengths for the tape-cast Al_2O_3/FeAlVCMn composites were nearly identical, Figure 13a. Despite a significant improvement in fiber strength for fibers etched from tape-cast FeNiCoCrAl composites, the fibers started failing at lower stresses during a tensile test in the tape-cast FeNiCoCrAl composite compared to the powder cloth fabricated composite, Figure 13b. For Al_2O_3/FeNiCoCrAl composites fabricated by both techniques, the fibers failed at lower stresses in the composite than in single fiber tensile tests. The Weibull moduli were also slightly lower for the AE analyzed data.

IV. DISCUSSION

The experimentally measured room temperature ultimate tensile strength, σ_{UTS}, for both composite systems has been compared to predictions based on Curtin's model given below[15] :

$$\sigma_{UTS} = V_f \left[\left(\frac{2}{m+2} \right)^{\frac{1}{m+1}} \left(\frac{m+1}{m+2} \right) \right] \left[\frac{\sigma_f^m \tau_f L_o}{r} \right]^{\frac{1}{m+1}} + \left(1 - V_f \right) \sigma_y$$

where V_f is the fiber volume fraction, *m* is the Weibull modulus, σ_f is the average etched-out fiber strength at gage length L_o, τ_f is the frictional shear stress, *r* is the fiber radius, and σ_y is the yield strength of the matrix. Al_2O_3/FeAlVCMn composite strength was slightly underestimated by Curtin's model,[15] with the composites achieving 98-119% of predictions, Table VI. However, Curtin's model very accurately predicted the strengths of both powder-cloth and tape-cast fabricated Al_2O_3/FeNiCoCrAl composites, Table VI. As observed experimentally, the model predicted equal strengths for the Al_2O_3/FeNiCoCrAl composites fabricated by the two methods even though a significant difference in etched-out fiber strength was measured. Using the model, the difference in Weibull moduli between the tape-cast and powder-cloth fabricated composites, Table III, was enough to offset the higher strength of the fibers etched from tape-cast fabricated composites. The Weibull moduli used in the predictions were based on 20 single fiber tests, usually a sufficient population size for estimating Weibull moduli.[16] However, the average in-situ strengths of the Al_2O_3 fibers, as measured by AE analysis, were essentially identical and this is probably the true reason the strength of the powder cloth and tape-cast FeNiCoCrAl composites achieved equal ultimate tensile strengths.

The in-situ strength of the Al_2O_3 fibers was lower than expected based on single fiber tests in the FeNiCoCrAl composites fabricated by both techniques, Figure 13b. The average strength of the single fiber tests may be slightly higher than in-situ fiber strength as a result of stronger fibers being inadvertently selected for single fiber tests due to the testing requirements. Fibers have to be a minimum of 60 mm in length to test. The weakest fibers may have broken during processing into segments shorter than 60 mm in length (Figure 3) and therefore, were not available to be tested. In addition to inadvertently testing higher strength fibers in single fiber tensile tests, the lower in-situ fiber strength is likely due to a combination of pre-existing fiber breaks from composite fabrication, bending stresses in the fiber induced by processing, larger volume of fiber being tested, and low interfacial shear strength between the fiber and the matrix. Pre-existing fiber breaks from composite fabrication could be acting as stress concentrators,[17-18] hence the fiber failures occur at low strains. Bending stresses can develop in the fibers during processing due to uneven distribution of matrix powder and result in a reduction in the additional load required to cause the fiber to fail.[19] Fiber failures at low strains are also more likely to occur in composites due to the large volume of fiber in a composite. The probability of a large flaw initiating fracture at lower stresses increases with the volume of fiber tested, especially with the large variation in fiber strength (low m). The low interfacial shear strength also contributes to the low strength of the Al_2O_3 fibers in FeNiCoCrAl. With a highly ductile matrix, the strength of a composite decreases with decreasing interfacial shear strength, especially with a large variation in fiber strength.[20-21] Interfacial shear strengths are directly related to the critical length of fiber, l_c. With weak interfacial shear strengths, the critical length of fiber is long and the load bearing efficiency of the fiber is reduced from the broken end to a distance of 0.5 l_c.[22] In contradiction with the Al_2O_3/FeNiCoCrAl composites, the fibers in the Al_2O_3/FeAlVCMn system had essentially equal strengths in single fiber tensile tests and in-situ during composite tensile tests.

The low Weibull moduli of the etched-out fibers, ranging from 4 to 9, is indicative of wide scatter in fiber strength. A wide scatter in fiber strength promotes cumulative failure,[18, 20] described as the gradual accumulation of fiber breaks prior to composite failure. Both Al_2O_3/FeAlVCMn and Al_2O_3/FeNiCoCrAl composites failed in a cumulative manner. As the weakest fibers failed, the neighboring fibers were strong enough to bear an increase in load until a significant number of fibers failed in a cross section and either failure or a load drop occurred.

The average strengths of fibers etched from both matrices were significantly increased compared to our previous study of Al_2O_3 fibers etched-out of FeCrAlY[2]. The strength of the fibers etched from tape cast FeAlVCMn and FeNiCoCrAl averaged 2400 ± 326 MPa and 2719 ± 250 MPa, respectively, whereas the strength of the fibers etched from FeCrAlY averaged only 1693 ± 180 MPa[2]. The fibers etched from FeAlVCMn matrix composites still degraded approximately 34% from the as-received fiber strength, Table III. This is similar to the percentage of strength degradation seen in the previous study of fibers etched from FeCrAlY.[2] However, the room temperature as-received strength of the Al_2O_3 fibers had increased considerably due to improvements in fiber processing, from 2484 ± 128 MPa (1990-91) to 3580 ± 126 MPa (1994-95). To determine if the improved etched-out fiber strength is simply a result of the improved as-received fibers or due to less reactive matrix compositions, recent vintage Al_2O_3 fibers were composited into a FeCrAlY matrix composites and etched-out. The etched-out strength averaged 1847 ± 189 MPa, not significantly different from the previous study. Therefore, the improvement in etched-out fiber strength was primarily due to using matrices with less reactive elements. However, the degradation in strength was still 24 % for the FeNiCoCrAl tape-cast composites and 33 % for the FeAlVCMn tape-cast composites. It is unclear at this time what is causing the strength reduction for the fibers etched from FeNiCoCrAl tape-cast composites. The surface of fibers etched from FeNiCoCrAl tape-cast composites were very smooth with no visible flaws. Chemical interaction between Al_2O_3 and FeNiCoCrAl was also shown to be negligible from the fiber push-out tests. Slight improvements in fiber strength may be possible with improvements in the binders used for composite fabrication, however, it is unlikely that Al_2O_3 fibers will maintain their strength after processing.

While the strength of etched-out fibers was significantly improved by the choice of matrices with less reactive elements, the room temperature ultimate tensile strengths of both composite systems were less than desired. The ultimate tensile strengths of both composite systems were reduced as a result of a low Weibull modulus for the etched-out Al_2O_3 fibers. Although the fibers etched from tape-cast FeNiCoCrAl matrix composites had higher strengths than fibers etched from FeAlVCMn, the UTS of the Al_2O_3/FeNiCoCrAl composites were considerably lower than the average strength of the Al_2O_3/FeAlVCMn composites. The low strength of the Al_2O_3/FeNiCoCrAl composites is attributed to the lower in-situ fiber strength compared to etched-out fiber strength, the low frictional shear stress between Al_2O_3 and FeNiCoCrAl, and the low yield strength of FeNiCoCrAl. A correlation of higher etched-out fiber strength to a lower interfacial bond strength exists for the three Fe-base alloy matrices studied, FeCrAlY,[2] FeAlVCMn, and FeNiCoCrAl. For ductile matrices, composite strength increases with increasing interfacial bond strength.[20-21] Therefore, further improvements in fiber strength retention may not improve the strength of the composites.

To further evaluate the feasibility of Al_2O_3/FeAlVCMn, the density compensated 800 °C strength of Al_2O_3/FeAlVCMn was estimated using Curtin's model and estimated elevated temperature properties. The density compensated 800 °C strength was estimated to be at the bottom of the range for wrought superalloys at 800 °C. Given the high cost of composite fabrication, the mechanical properties of these composite systems do not justify a continued development effort.

V. SUMMARY OF RESULTS

1. The strength of etched-out Al_2O_3 fibers was significantly improved by the choice of matrices with less reactive elements. In comparison to fibers etched from matrices in previous studies,[1-4] the fiber surfaces were smooth and undamaged. However, a minimum of 24% degradation from as-received fiber strength was still observed.
2. Powder-cloth and tape-casting fabrication techniques resulted in similar microstructures for both composite systems. Etched-out fiber strengths were equivalent for the FeAlVCMn matrix using both fabrication techniques but powder-cloth processing resulted in 14% more fiber strength degradation for FeNiCoCrAl.
3. The ultimate tensile strengths and elastic moduli of the composites were higher than their respective monolithics at the expense of material ductility.
4. The Weibull plot of strength values for fibers etched from Al_2O_3/FeAlVCMn was identical to the Weibull plot containing strengths calculated from acoustic emission data during a composite tensile test. However, the in-situ strengths of Al_2O_3 fibers in FeNiCoCrAl was less than expected based on etched-out fiber strengths.
5. The UTS of Al_2O_3/FeNiCoCrAl was considerably lower than the strength of Al_2O_3/FeAlVCMn. The Al_2O_3/FeNiCoCrAl had low in-situ fiber Weibull modulus, low frictional shear stress between the fiber and matrix, and low matrix yield strength.

6. The UTS of Al_2O_3/FeAlVCMn was equal to or higher than predicted by Curtin's model. The model accurately predicted the strength of the Al_2O_3/FeNiCoCrAl composites.

VI. CONCLUSIONS

Single-crystal Al_2O_3 fibers can be processed with less fiber strength degradation by the selection of non-reactive matrices. However, the ultimate tensile strengths of the resulting composites were less than desired due to low fiber Weibull moduli. Additionally, the strength of the Al_2O_3/FeNiCoCrAl composite was degraded due to the Al_2O_3 fiber having a lower than expected in-situ strength and a low interfacial bond strength between the FeNiCoCrAl matrix and Al_2O_3 fiber. The room temperature and estimated elevated temperature strengths of both composite systems were not sufficient to continue development efforts. Acoustic emission analysis during tensile testing was a useful tool for determining the efficiency of the fibers in the composite and for determining the failure mechanism of the composites.

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Table I. Alloy Compositions, at. %

	Fe	Ni	Co	Cr	Al	Nb	V	C	Mn	Ti	Y
FeAlVCMn	75.6				21.3		1.47	1.19	0.46		
Thermo-span®	35.3	24.6	28.4	6.1	1.1	3.0				1.0	
FeNiCoCrAl	40.7	24.2	28.0	6.0	1.1						
FeCrAlY	68.0			24.0	8.0						0.06

Table II. Interstitial Content of Composite Plates

Matrix	Fab. Technique	C, ppm	F, ppm	N, ppm
FeAlVCMn	Powder Cloth		11000	397
FeAlVCMn	Tape Cast		<10	360
FeNiCoCrAl	Powder Cloth	310	33	323
FeNiCoCrAl	Tape Cast	106	<10	455

Table III. Tensile Strength of Al_2O_3 Etched from Composite

Matrix	Fabrication Technique	Average σ_f (MPa)	Weibull Modulus, m	Minimum σ_f (MPa)
FeAlVCMn	Powder Cloth	2322 ± 232	9.0	1237
FeAlVCMn	Tape Cast	2400 ± 326	5.3	1103
FeNiCoCrAl	Powder Cloth	2203 ± 136	5.3	1783
FeNiCoCrAl	Tape Cast	2719 ± 250	3.7	1475
As-received		3580 ± 126	15.7	3043

Table IV. Debond and Frictional Shear Strengths

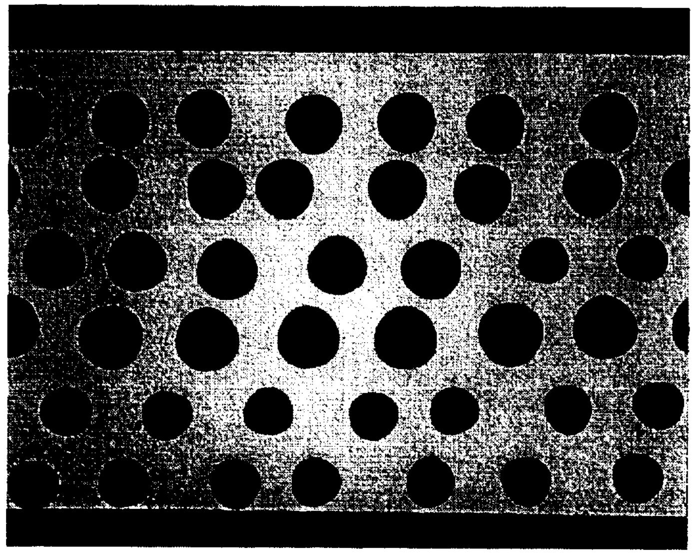
Sample	Number of Tests	τ_d (MPa)	τ_f (MPa)
Al_2O_3/FeAlVCMn	20	158 ± 15	120 ± 15
Al_2O_3/FeNiCoCrAl	20	30 ± 3	21 ± 3

Table V. Tensile Properties of Monolithics

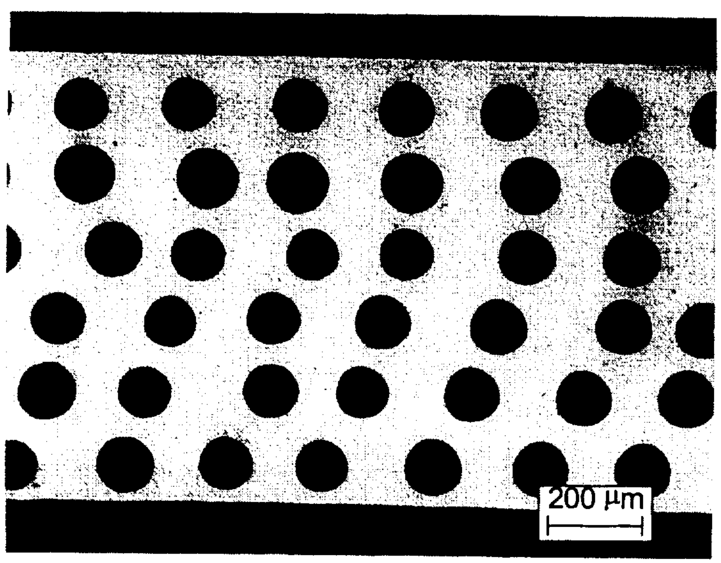
Material	σ_{UTS} (MPa)	$\sigma_{0.2\%y}$ (MPa)	E (GPa)	ε_f (Pct)
FeAlVCMn	817 ± 29	762 ± 23	163 ± 16	1.20 ± 0.48
FeNiCoCrAl	464 ± 56	254 ± 5	157 ± 27	>10

Table VI. Tensile Properties of Composites

Material	σ_{UTS} (MPa)	σ_{el} (MPa)	E (GPa)	ε_{UTS} (Pct)	ε_{el} (Pct)	V_f (Pct)	Predicted[15] UTS (MPa)
Al_2O_3/FeAlVCMn							
- Tape Cast	989	383	238	0.57	0.18	26	972
	1148	391	228	0.71	0.17	33	1028
	1152	341	249	0.72	0.13	26	972
Al_2O_3/FeNiCoCrAl							
- Powder Cloth	638	214	258	0.38	0.14	33	640
	624	138	215	0.52	0.06	33	640
- Tape Cast	636	182	207	0.41	0.10	34	646
	674	247	254	0.38	0.11	34	646



(a)



(b)

Figure 1. Microstructure of Al_2O_3/FeNiCoCrAl Composites (a) powder-cloth and (b) taper-cast.

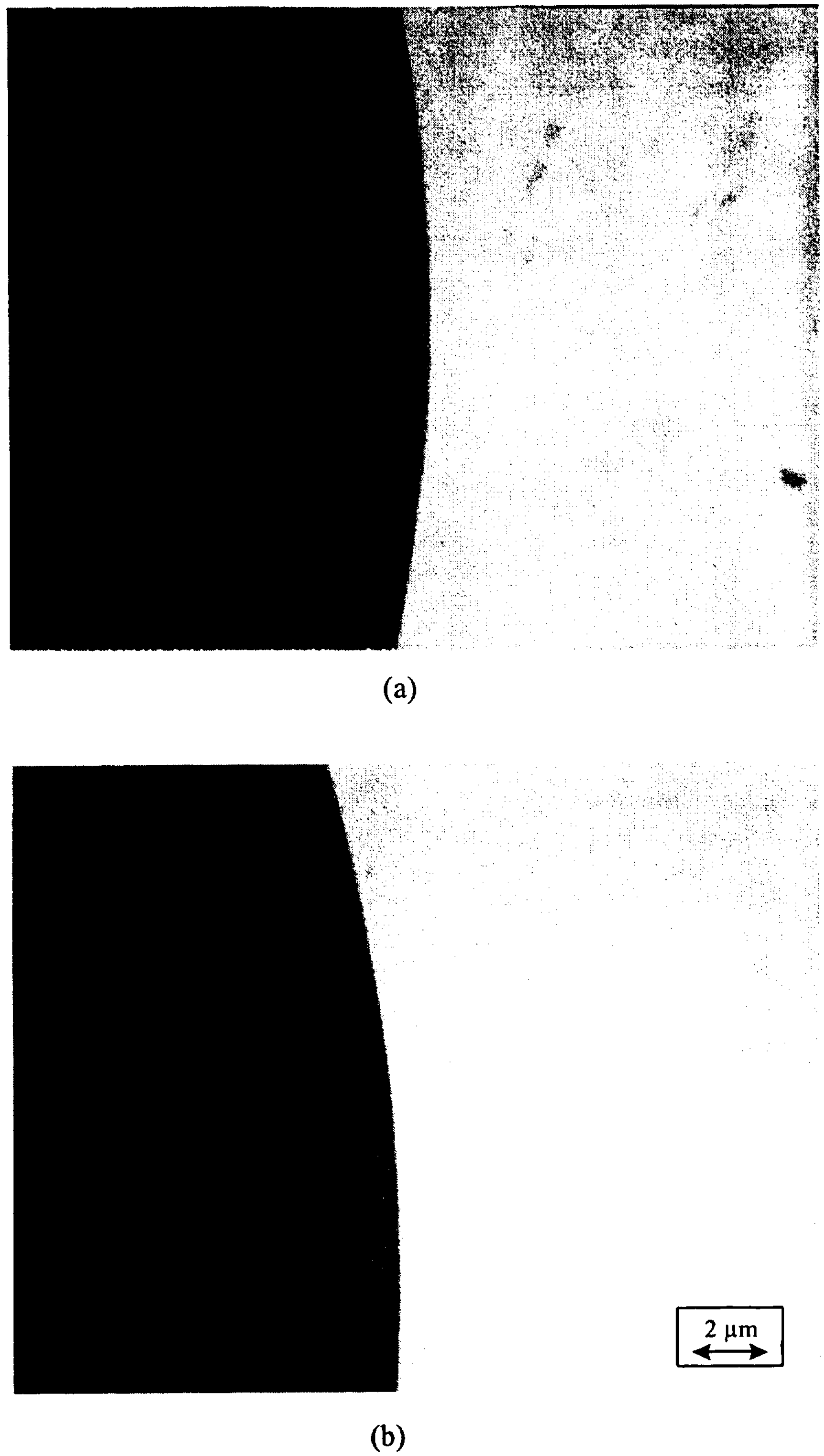
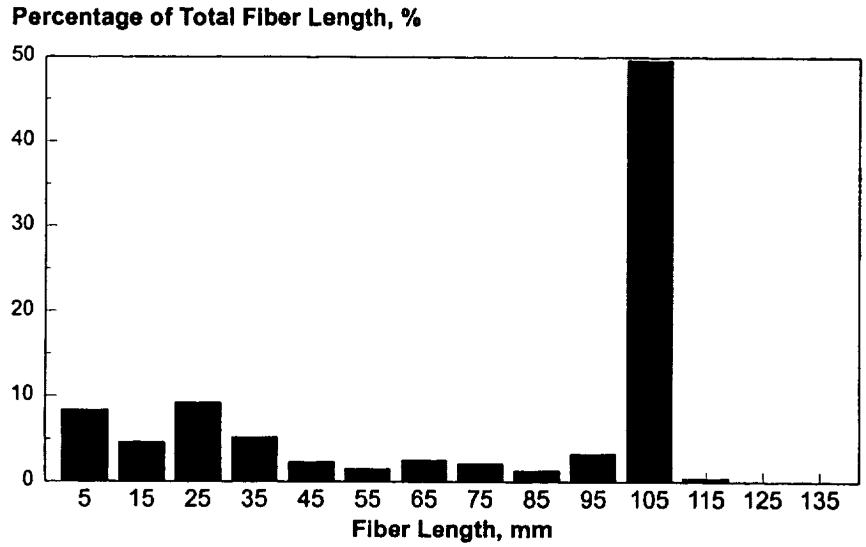
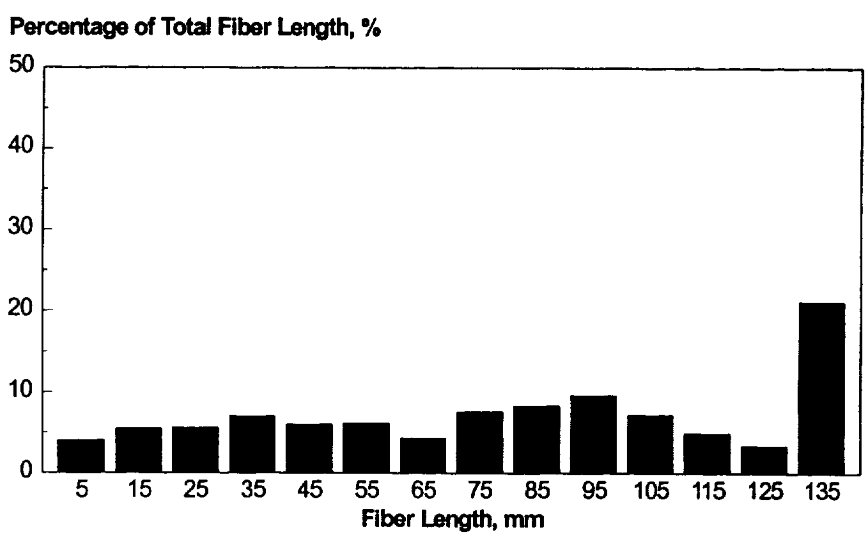


Figure 2. Interface between Al_2O_3 and (a) FeAlVCMn and (b) FeNiCoCrAl.



(a)



(b)

Figure 3. Distribution of Al_2O_3 fiber lengths etched from tape-cast (a) FeAlVCMn and (b) FeNiCoCrAl matrix composites.

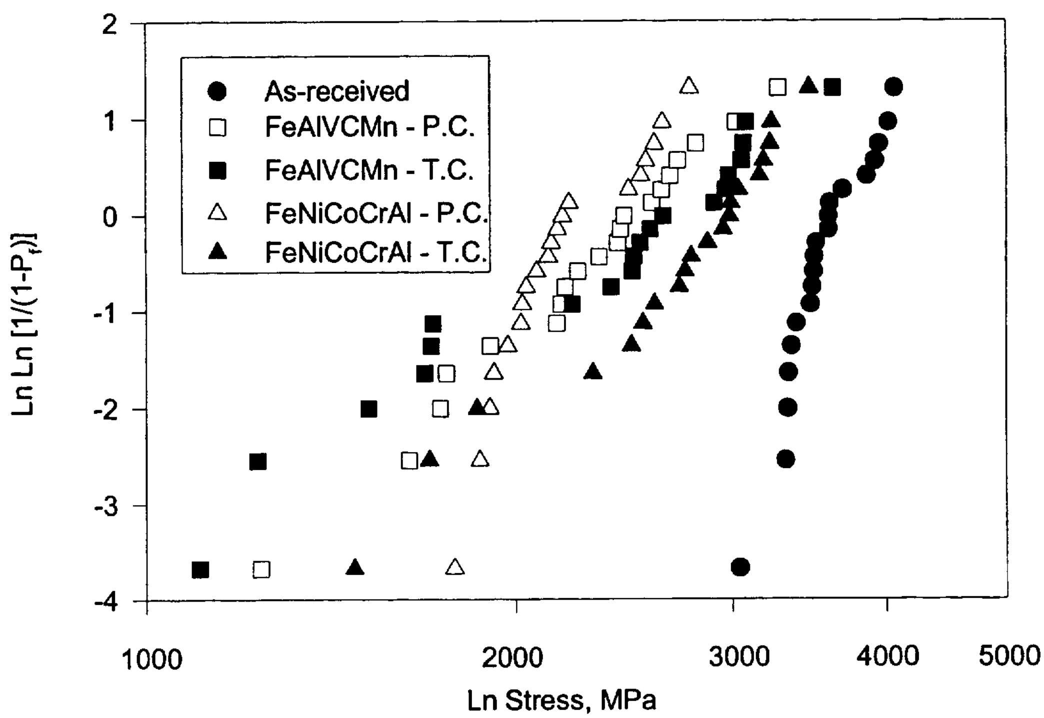


Figure 4. Weibull plot of Al_2O_3 fiber strengths in the as-received condition and after etching from powder-cloth (P.C.) and tape-cast (T.C.) composites.

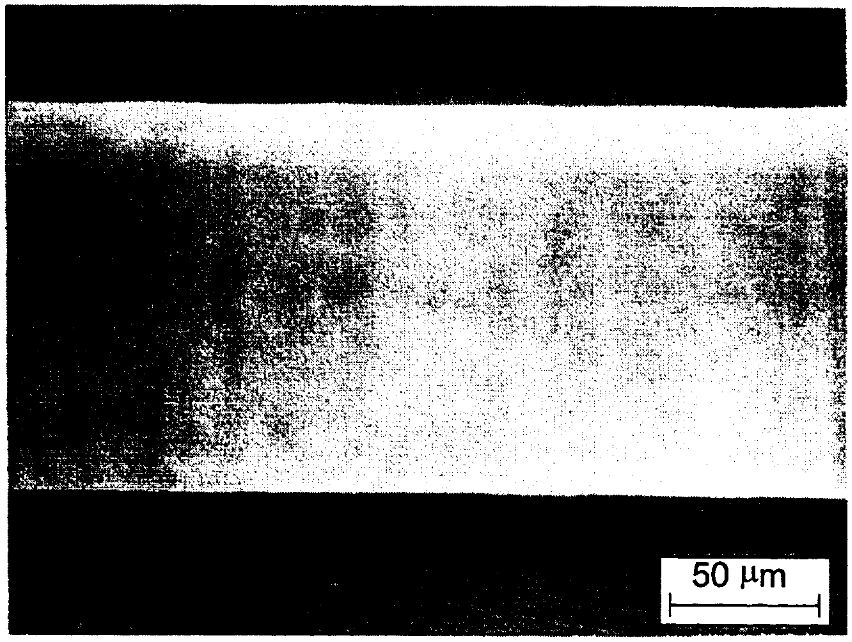
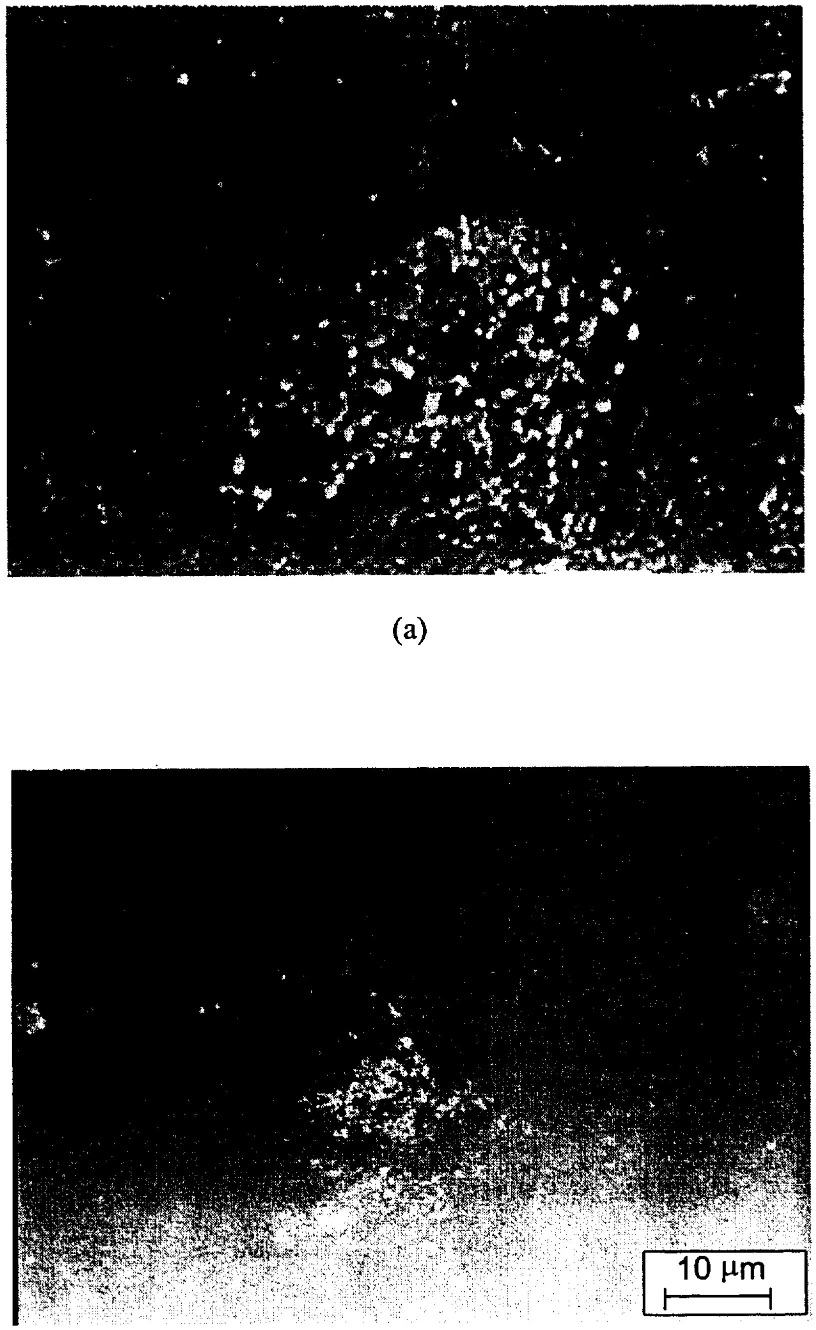


Figure 5. Surfaces of Al_2O_3 fibers etched from tape-cast FeAlVCMn composites were fairly smooth and undamaged.



(a)

(b)

Figure 6. Surfaces of Al_2O_3 fibers etched from (a) powder cloth and (b) tape-cast fabricated FeNiCoCrAl composites.

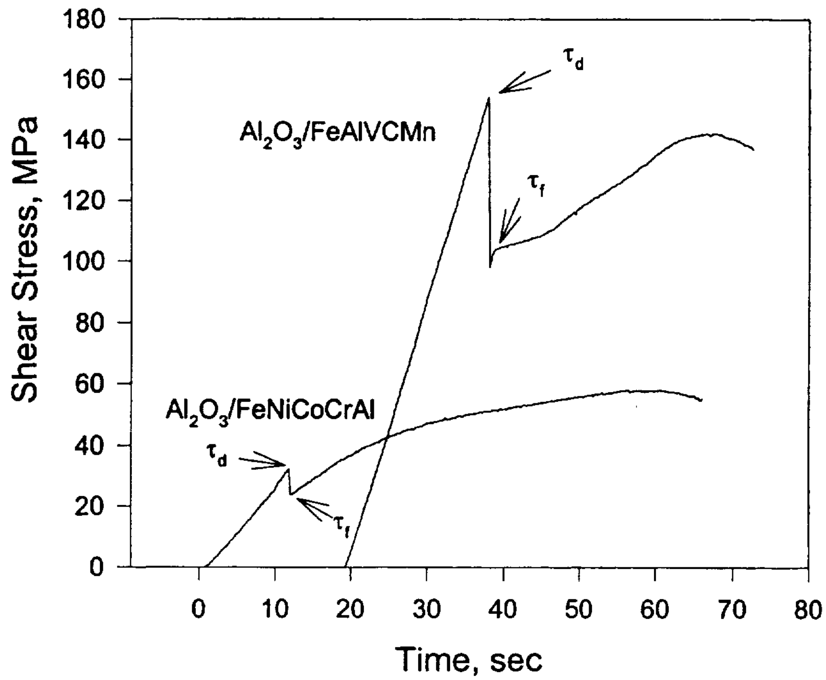


Figure 7. Typical room temperature stress-time plots for fiber push-out tests.

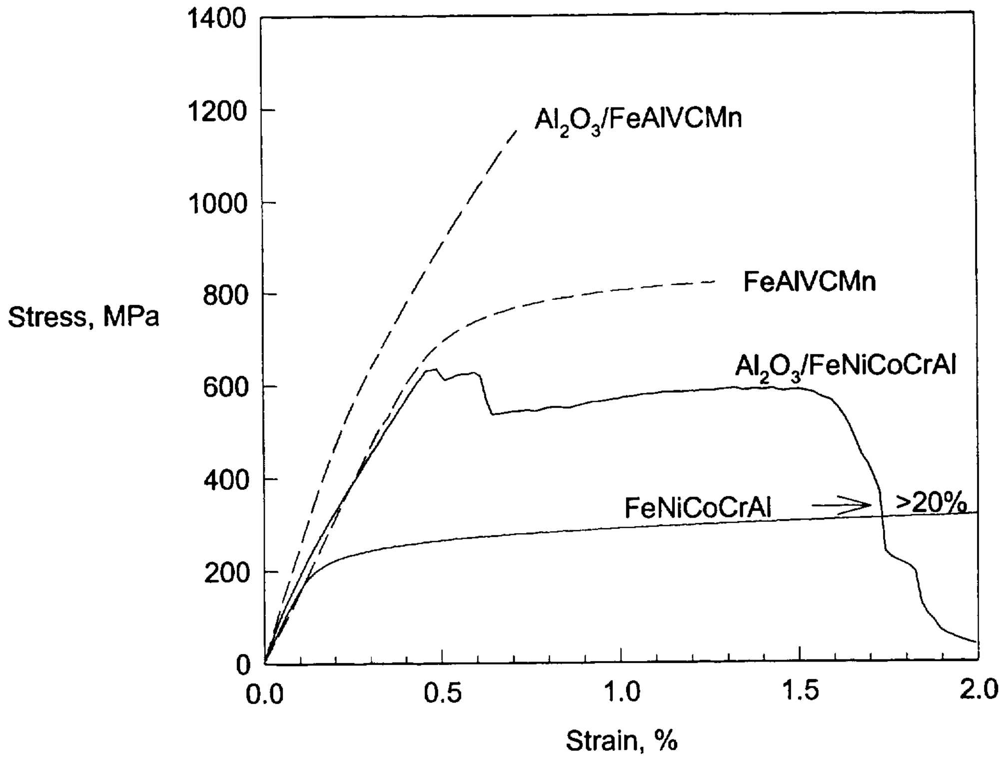
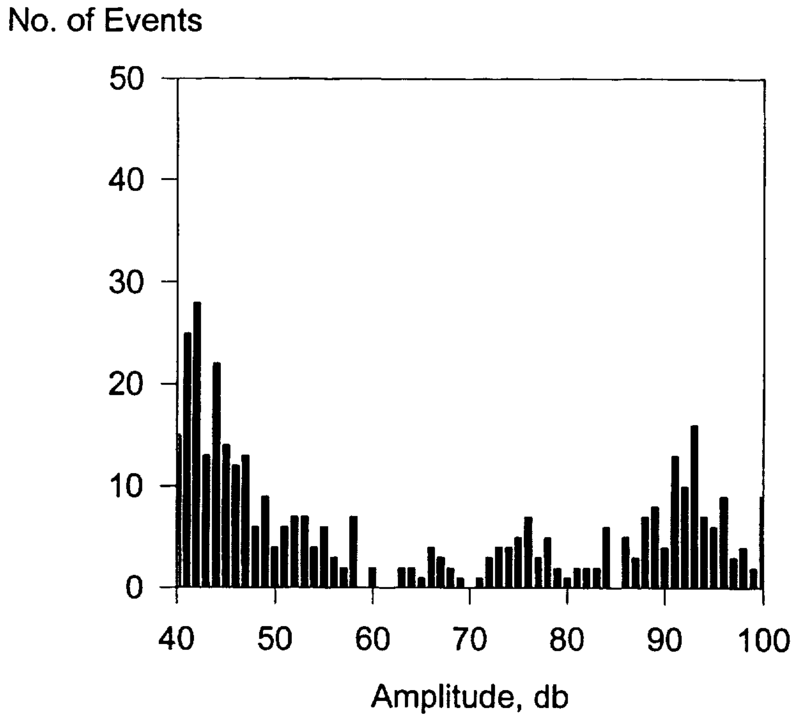
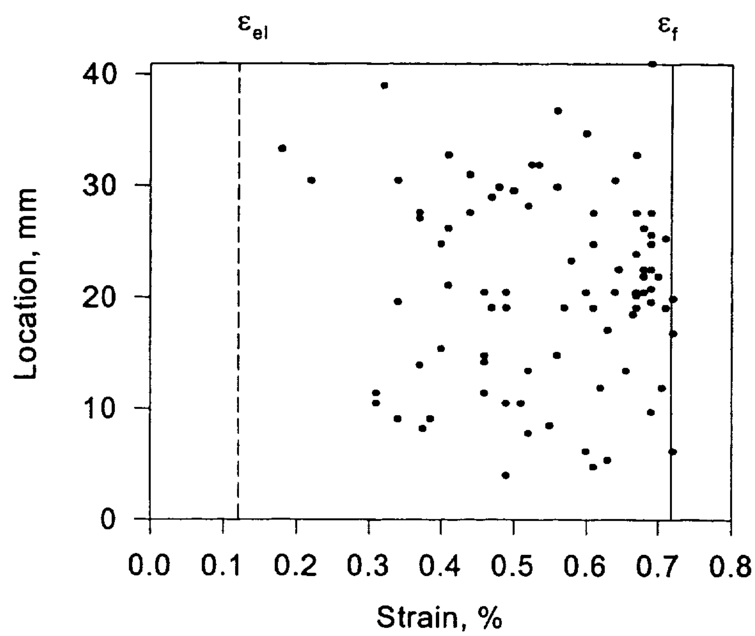


Figure 8. Typical monolithic and composite stress-strain curves.

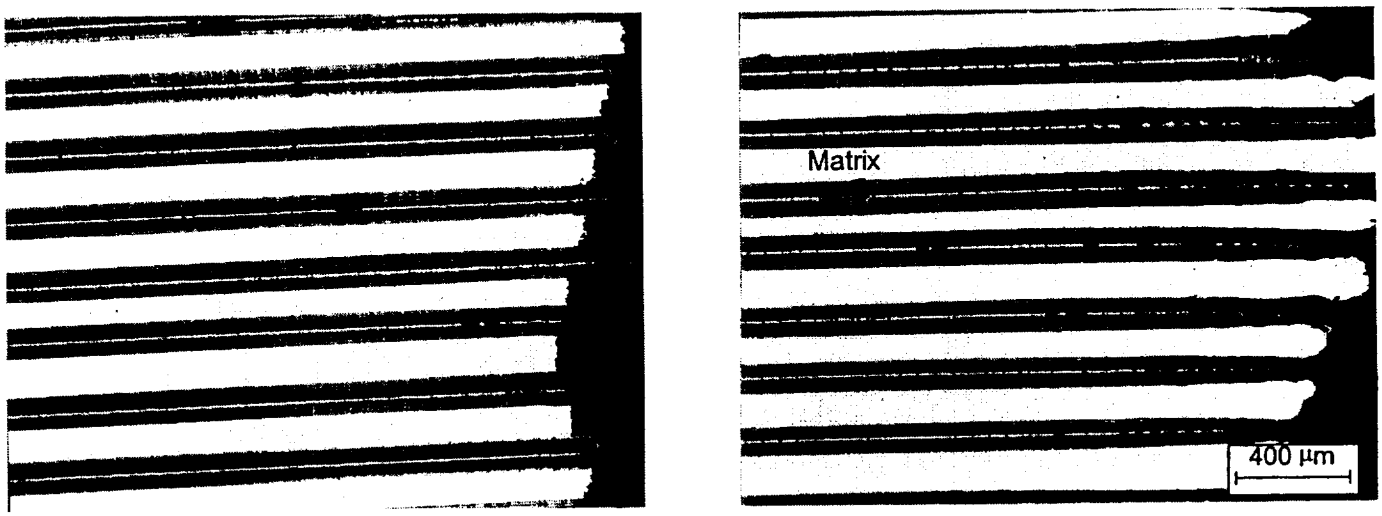


(a)



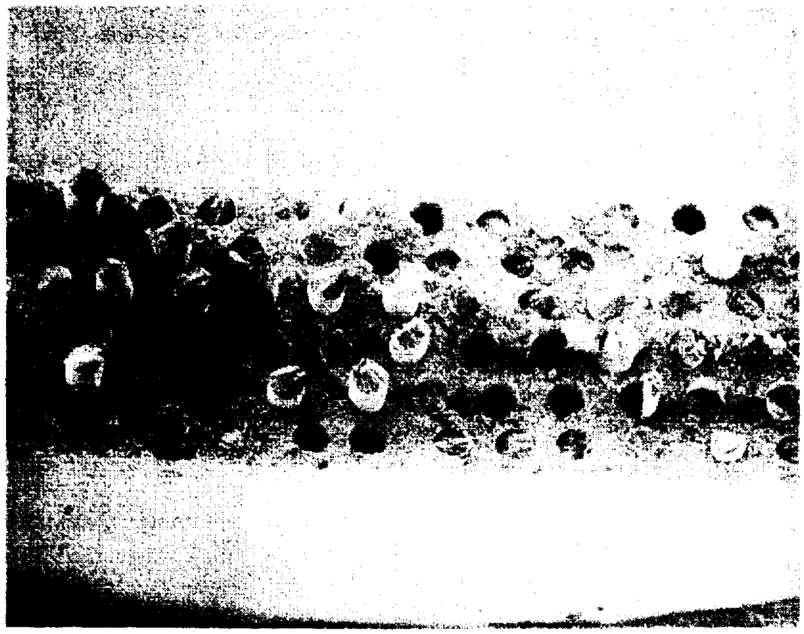
(b)

Figure 9. (a) Histogram of acoustic emission events during tensile test of Al_2O_3/FeAlVCMn. (b) Source location plot of acoustic emission events with amplitudes greater than 87 db.

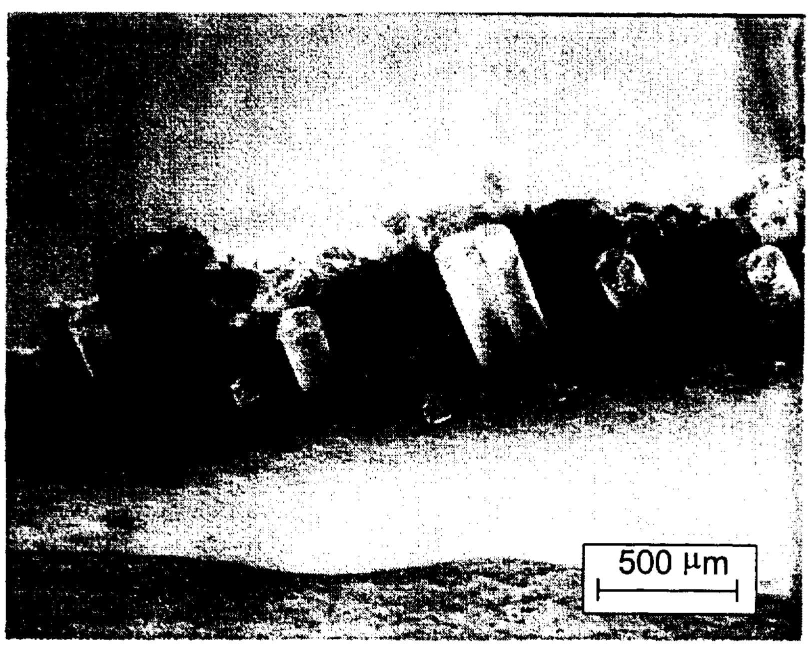


(a) (b)

Figure 10. Longitudinal sections of (a) Al_2O_3/FeAlVCMn and (b) Al_2O_3/FeNiCoCrAl fractured tensile specimens showing fiber fractures close to the fracture surface.

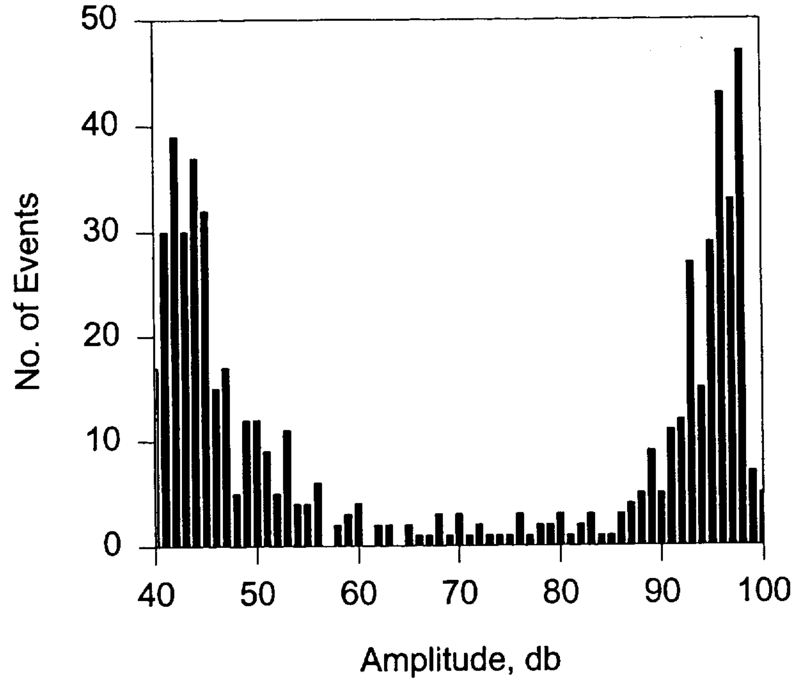


(a)

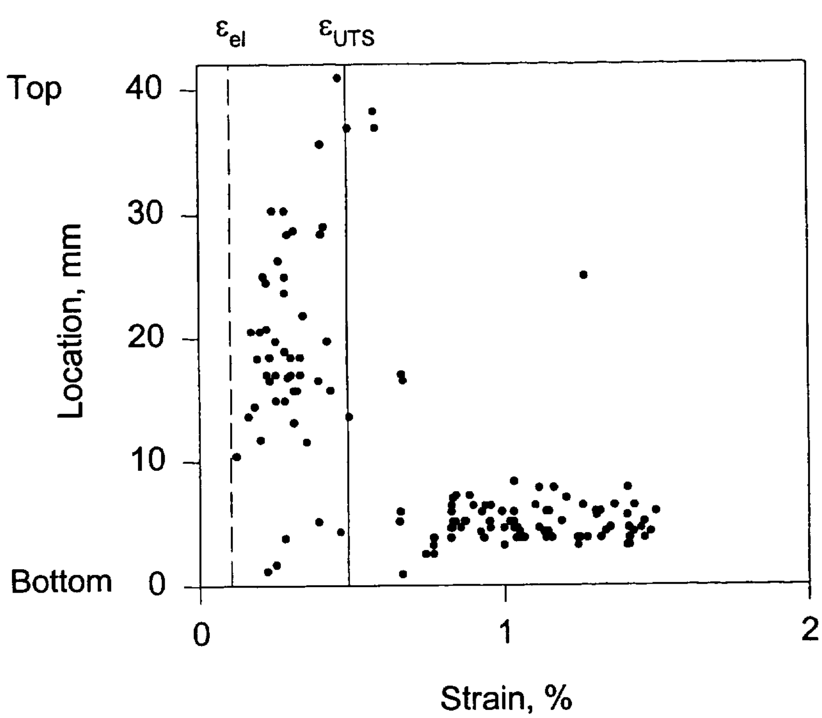


(b)

Figure 11. Fracture surfaces of (a) Al_2O_3/FeAlVCMn exhibited little fiber pullout while (b) Al_2O_3/FeNiCoCrAl showed extensive pullout.



(a)



(b)

Figure 12. (a) Histogram of acoustic emission events during tensile test of Al_2O_3/FeNiCoCrAl. (b) Source location plot of acoustic emission events with amplitudes greater than 87 db.

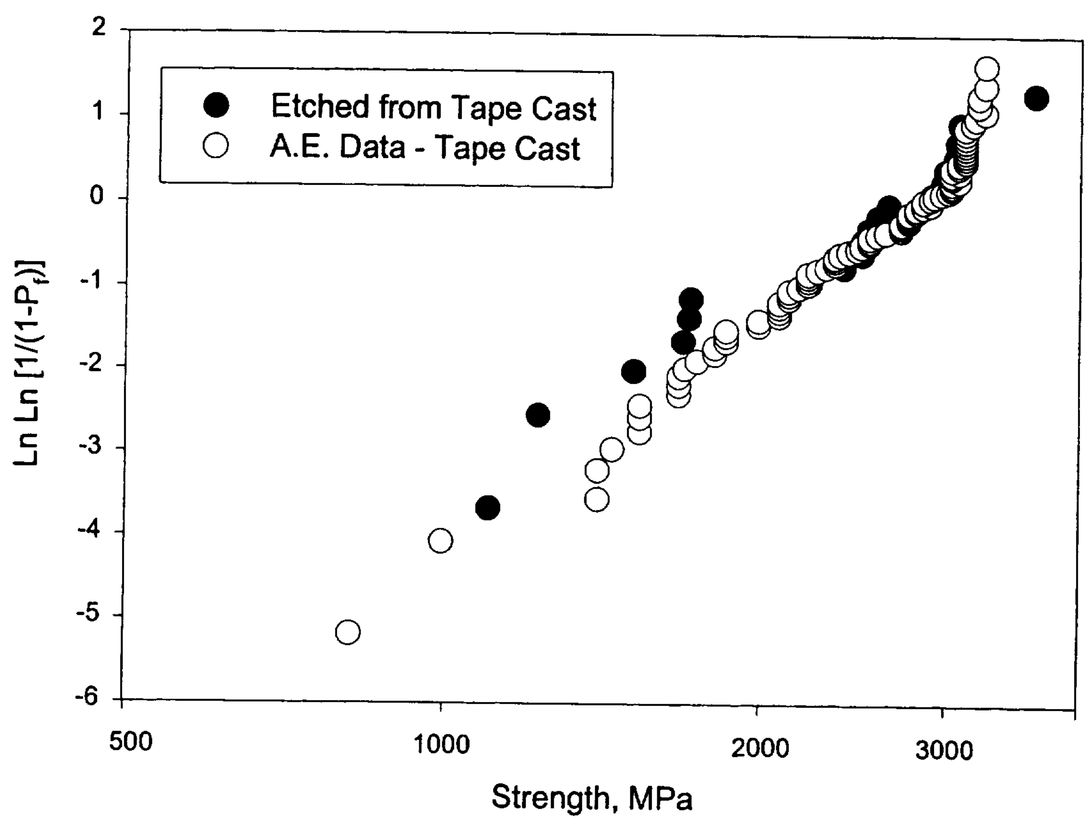


Figure 13a - Weibull plot comparing in-situ Al_2O_3 fiber strength to etched from composite fiber strength for tape-cast fabricated FeAlVCMn composites.

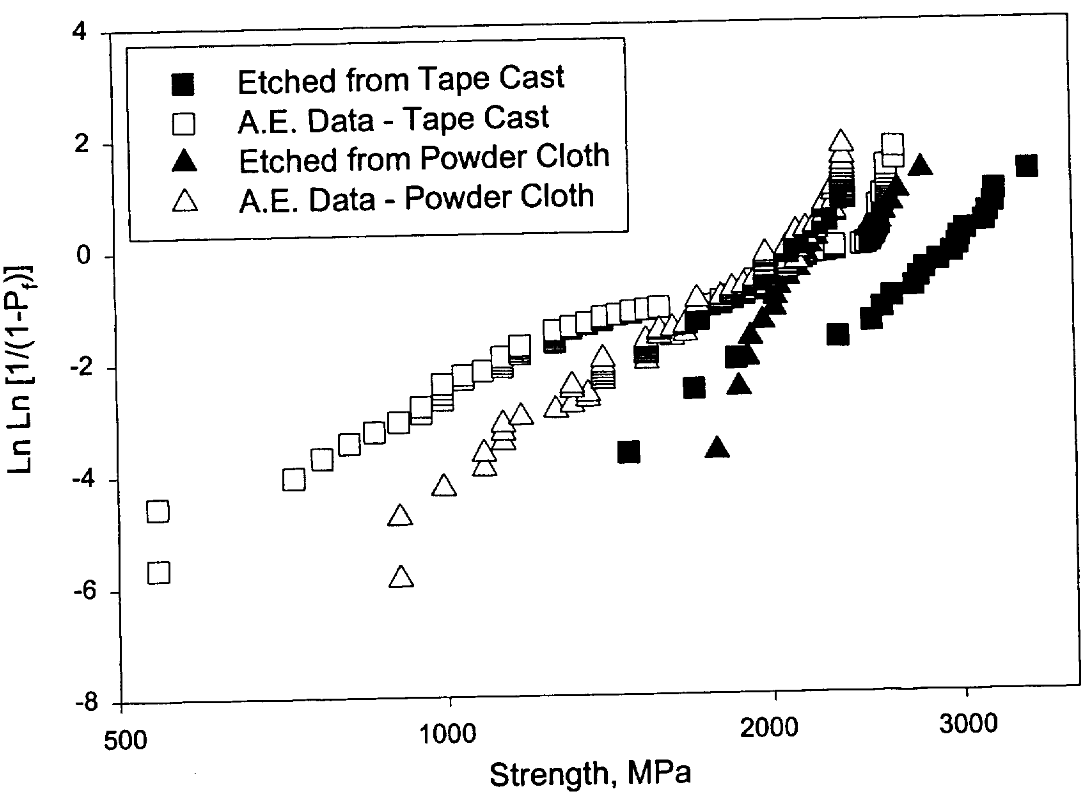


Figure 13b - Weibull plot comparing in-situ fiber strength to etched from composite fiber strength for both tape-cast and powder cloth fabricated FeNiCoCrAl matrix composites.